भारतीय मानक **IS 11960 : 2023**

Indian Standard

कठोर धातुओं में स्पष्ट सरंध्रता और असंयुक्त कार्बन के मेटालोग्राफिक का निर्धारण — पद्धति

(पहला पुनरीक्षण)

Metallographic Determination of Apparent Porosity and Uncombined Carbon in Hardmetals — Method

(First Revision)

ICS 77.160

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

December 2023 **Price Group 6**

Powder Metallurgical Materials and Products Sectional Committee, MTD 25

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Powder Metallurgical Materials and Products Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1987. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

In addition, the following changes have been made:

a) Reference clause has been added;

b) In **5**, calibration of measurement apparatus is included;

c) In **6**, characterisation of porosity and carbon (graphite) defects and characterisation of η - phase (eta-phase) has been added;

d) In **7**, determination of carbon defects and determination of the η - phase (eta-phase) has been added;

e) Test report clause has been modified; and

f) Figures have been updated.

In the preparation of this standard, assistance has been derived from the following standard ISO 4499-4 : 2016 'Hardmetals — Metallographic determination of microstructure — Part 4: Characterisation of porosity, carbon defects and eta-phase content'.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard

METALLOGRAPHIC DETERMINATION OF APPARENT POROSITY AND UNCOMBINED CARBON IN HARD METALS — METHOD

(First Revision)

1 SCOPE

This standard specifies the procedure for the metallographic determination of the presence, type and distribution of porosity and uncombined carbon in hardmetals.

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	*Title*
IS 11520 : 1985	Method for metallographic sample preparation of hardmetals
IS 11959 : 1987	Method for metallographic determination of microstructure in hardmetals

3 TERMINOLOGY

3.1 Carbon Defects — Macroscopic precipitates of carbon (graphite) which can be in the form of large angular rosettes or small flakes.

3.2 Eta-Phase/η - Phase — Cubic carbide based on M6C or M12C structure where M is a mixture of Co and W usually in equal proportions, and which can be present as large (up to 100 μm diameter) rosettes or small micrometres.

4 APPARATUS

4.1 Metallographic Optical Microscope

Metallographic optical microscope or other suitable equipment permitting observations and measurements up to a magnification of 200 X.

4.2 Scanning Electron Microscope (SEM)

It permitting observation and measurement of features too small to be resolved with optical microscope.

4.3 Equipment

Equipment for the metallographic preparation of test specimens.

5 CALIBRATION OF MEASUREMENT APPARATUS

5.1 For reliable quantitative measurements, images shall be calibrated against a stage micrometre or scale traceable to a National Reference Standard.

5.2 For images obtained from an optical microscope, an image of the calibration graticule shall also be obtained using the same objectives (and internal magnification step changers or zoom position) and illuminating technique. The microscope shall be set up for Kohler illumination to obtain the maximum resolution.

5.3 For images obtained from a scanning electron microscope, images of the graticule should be obtained under the same conditions (accelerating kV, working distance, illumination aperture) as those used for the hardmetals.

6 SPECIMEN PREPARATION

6.1 The specimen shall be selected and prepared for metallographic examination as per IS 11520.

6.2 The surface to be examined shall be unetched and free from grinding and polishing marks. Care shall be taken to avoid tearing out of particles, which may lead to a wrong evaluation of porosity.

6.3 Characterisation of Porosity and Carbon (Graphite) Defects

For porosity and carbon defects, the test-piece section shall be prepared as for metallographic examination and the surface to be examined shall be free from grinding and polishing marks. Care shall be taken to avoid tearing out of particles, which can lead to a misleading evaluation of porosity.

6.4 Characterisation of Eta-Phase

Etching is necessary to reveal eta-phase particles (*see* **7.5**). Eta-phase is metal carbide (usually M_6C or $M_{12}C$, where M is a combination of Co and W, for example, Co_3W_3C), that forms when the overall carbon content of the hardmetal is relatively low.

Generally, it can grow in one of two morphologies, either as large rosettes or as small particles of a similar size to the other hard phases (WC or cubic carbides) present in the hardmetal (*see* **7.5**). The presence of the eta-phase is usually determined after light etching in 10 percent Murakami's reagent for a few seconds with immediate water flush following etching (*see* IS 11959), which works well for identifying large eta-phase rosettes. When the eta-phase is present as smaller particles (*see* Fig. 6), it is recommended to use a 5 percent Murakami's solution for 20 min followed by washing the sample with water. In both cases, the surface should be dried carefully with acetone or alcohol without wiping.

7 PROCEDURE

7.1 Pore size is defined as the maximum dimension of the pore in the section. Special reference shall be made to the presence of cracks or slits.

7.2 Pore up to 10 µm shall be assessed by scanning the surface of the test piece section at a magnification of either 100 X or 200 X. An area fully representative of the test piece section shall be examined and compared with the range of photomicrographs shown in Fig. 1 or Fig. 2, according to the chosen magnification. The porosity level shall be reported by reference to the appropriate photomicrograph and designated as A02, A04, A06 or A08. If the level of A-type pores is less than 50 percent of that shown in Fig. 1 (A02) or Fig. 2 (A02), it shall be designated as A00.

7.3 Pores within the range 10 µm to 25 µm shall be assessed by scanning the surface of the test piece section at a magnification of 100 X. An area fully representative of the test piece section shall be examined and shall be compared with the range of photomicrographs shown in Fig. 3. The porosity level shall be reported by reference to the appropriate photomicrograph and designated as B02, B04, B06 or B08.

7.3.1 If the number of B-pores appears to be less than or equal to that represented by B02, the number of B-pores in the representative area (≥ 0.25 cm^2) shall be counted. This number shall be divided by the area examined to obtain the number of B-pores/cm^2.

7.3.2 If this number is less than 70 pores/cm^2, it shall be designated as B00 - #, where # is the number of B-pores/cm^2 so obtained. If the number is greater than or equal to 70 pores/cm^2, it shall be designated as B02.

7.3.3 If it is necessary to inspect for pores larger than 25 µm, they shall be examined at a suitable magnification up to 100 X and shall be counted and reported as the number of pores per unit area. The size ranges shall be chosen as follows: 25 µm to 75 µm, 75 µm to 125 µm, over 125 µm.

7.4 Determination of Carbon Defects

7.4.1 Uncombined carbon shall be assessed by scanning the surface of the test-piece section at a magnification of 100 X. An area fully representative of the test piece section shall be examined and shall be compared with the range of photomicrographs shown in Fig. 4. The level of uncombined carbon shall be reported by reference to the appropriate photomicrographs and designated as C02, C04, C06 or C08.

7.4.2 If A or B type porosity or C type uncombined carbon is not detected, it shall be reported as A00, B00 or C00 as applicable.

7.4.3 If the porosity or uncombined carbon is not uniform over the area of the test piece section being examined, the locations on the section to which the evaluation refers must be identified, for example as top, bottom, edge, rim (case), core, etc.

7.5 Determination of the η - Phase (Eta-Phase)

7.5.1 The eta-phase is a metal carbide (usually M_6C or $M_{12}C$, where M is a combination of Co and W, for example, Co_3W_3C), that forms when the overall carbon content of the hardmetal is relatively low. Generally, it can grow in one of two morphologies, either as large rosettes (*see* Fig. 5) or as small particles of a similar size to the other hard phases (WC or cubic carbides) present in the hardmetal (*see* Fig. 6). Phase of the eta-type appear orange to brown when observed by optical microscopy after light etching in 10 percent Murakami's reagent. Examine the whole surface at low magnification to reveal large rosettes (*see* Fig. 5) and then at progressively higher magnifications to observe smaller particles or grains. When the eta-phase is present as smaller particles, it is recommended to use a 5 percent Murakami's solution and the eta-phase is revealed as small white particles that contrast with the grey WC grains (*see* Fig. 6). A typical micrograph showing these smaller eta-phase particles is given in Fig. 6. The commonly used etching methods are listed in **6.4**. However, smaller particles cannot be identified easily using this method. SEM techniques such as atomic number contrast or orientation imaging microscopy (OIM) using electron back scatter diffraction techniques (EBSD) are more appropriate.

7.5.2 The presence of the eta-phase is usually determined after light etching in 10 percent Murakami's reagent for a few seconds with an immediate water flush following etching. The surface should be dried carefully with acetone or alcohol without wiping. Phases of the eta-type

appear orange to brown when observed by optical microscopy. Examine the whole surface at low magnification to reveal large rosettes (*see* Fig. 5) and then at progressively higher magnifications to observe smaller particles or grains. When the eta-phase is present as smaller particles (*see* Fig. 6), it is recommended to use a 5 percent Murakami's solution for 20 min. The eta-phase is revealed as small white particles that contrast with the grey WC grains. A typical micrograph showing these smaller eta-phase particles is given in Fig. 6.

7.6 Uncertainty of Measurement

Systematic and random measurement errors shall be avoided wherever possible.

8 TEST REPORT

8.1 The test reports shall include the following information:

a) Reference to this standard IS 11960;
b) Complete identification of the test specimen;
c) The results obtained;
d) Etchant and etching time;
e) Traceability, calibration graticule number and calibration certificate;
f) Imaging technique: optical or SEM techniques;
g) Magnifications used;
h) Number of fields of view measured;
j) For eta-phase characterisation, total number of intercepts;
k) Arithmetic mean linear intercept size for eta-phase characterisation in micrometres (μm);
m) All operations not specified in this part of IS 11960 are regarded as optional; and
n) Details of any occurrence that might have affected the result.

8.2 The test report should additionally include the following information:

a) Identification number of the image or photo micrographs if archived;
b) Information about the source of the material and the customer requesting the measurement to be made;
c) Numerical aperture of objective for optical microscopy;
d) Acceleration voltage, working distance and illuminating aperture for SEM; and
e) Comment on measurement uncertainty.

A02

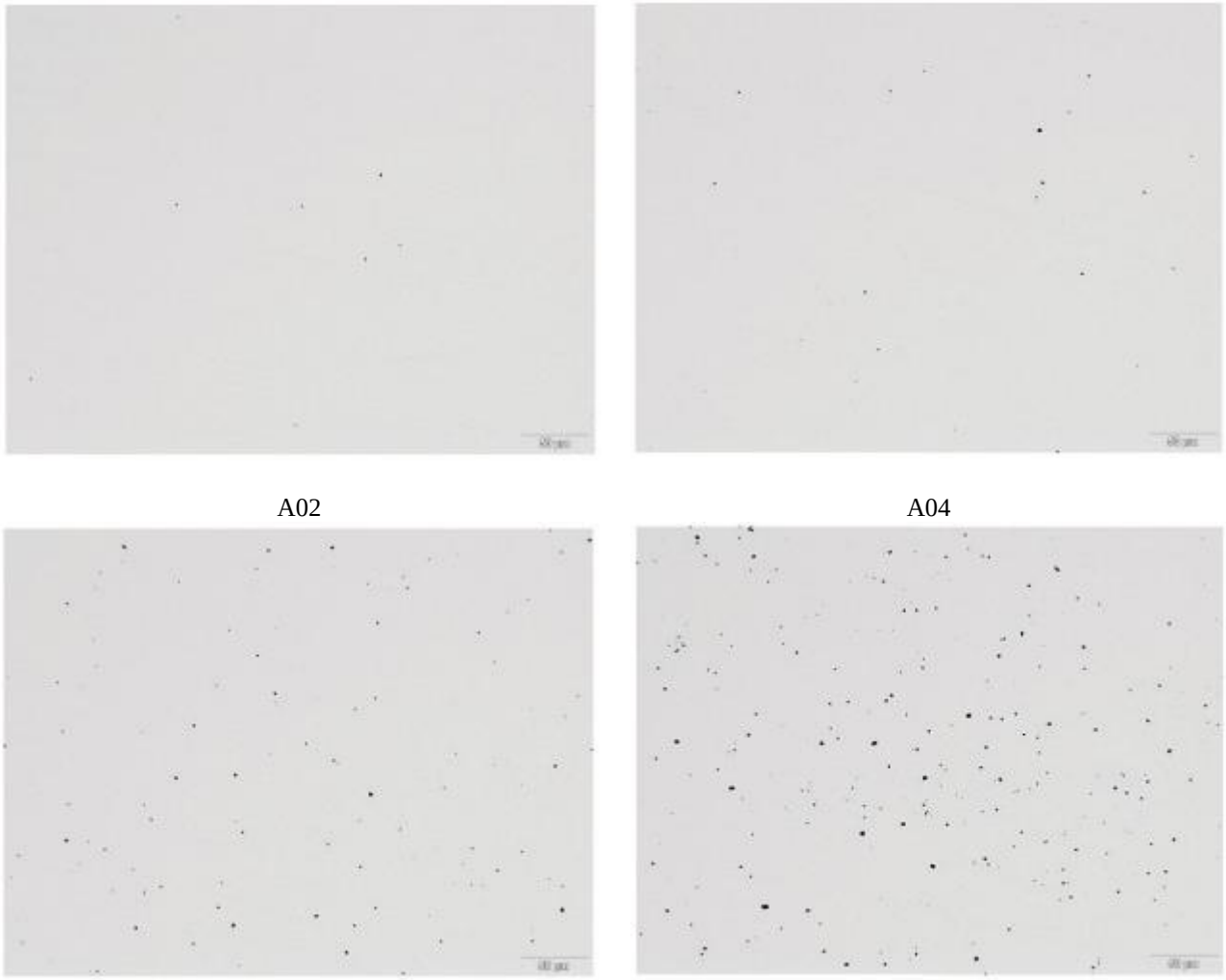
A04

A06

A08

FIG. 1 A-TYPE APPARENT POROSITY (100 X)

(THIS FIG.1 IS REPRODUCED FROM ISO 4499-4)



A02 A04

A06 A08

FIG. 2 A-TYPE APPARENT POROSITY (200 X)

(THIS FIG.2 IS REPRODUCED FROM ISO 4499-4)

B02

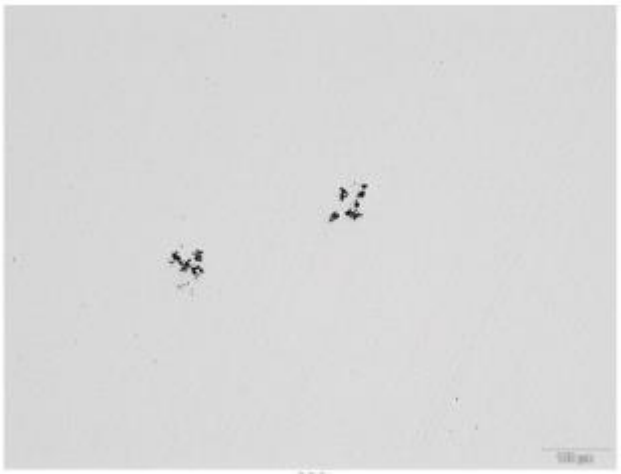
B04

B06

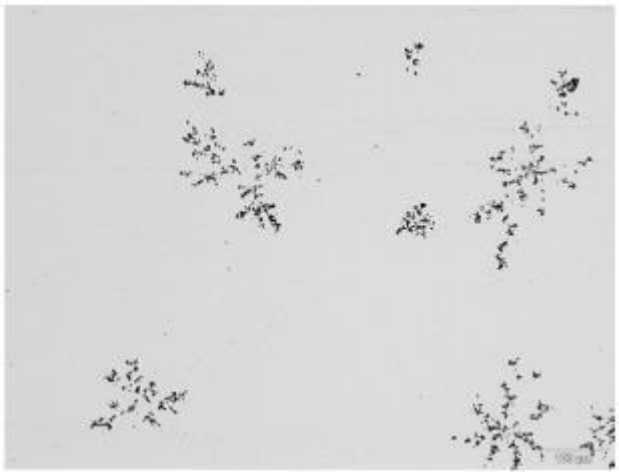
B08

FIG. 3 B-TYPE APPARENT POROSITY (100 X)

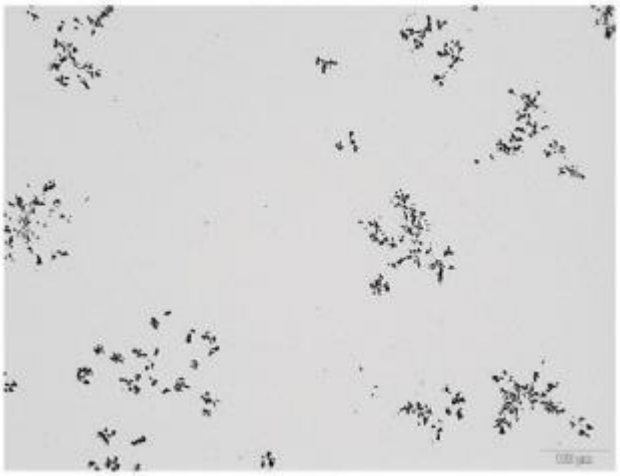
(THIS FIG.3 IS REPRODUCED FROM ISO 4499-4)



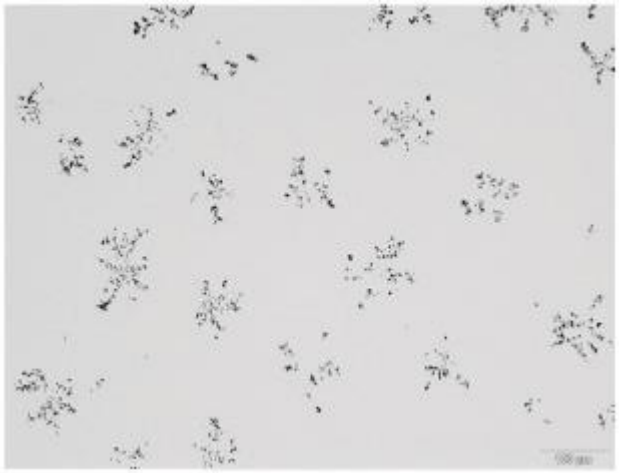
C02



C04



C06



C08

FIG. 4 UNCOMBIND CARBON (100 X)

(THIS FIG.4 IS REPRODUCED FROM IS0 4499-4)

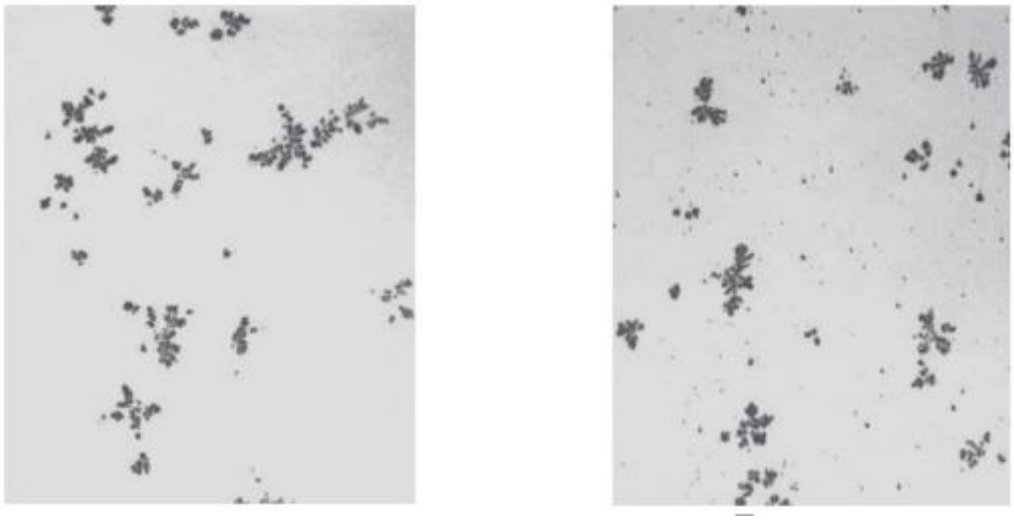


FIG. 5 LARGE ETA PHASE ROSETTES (ABOUT 100 µm IN DIAMETER)

(THIS FIG.5 IS REPRODUCED FROM ISO 4499-4)

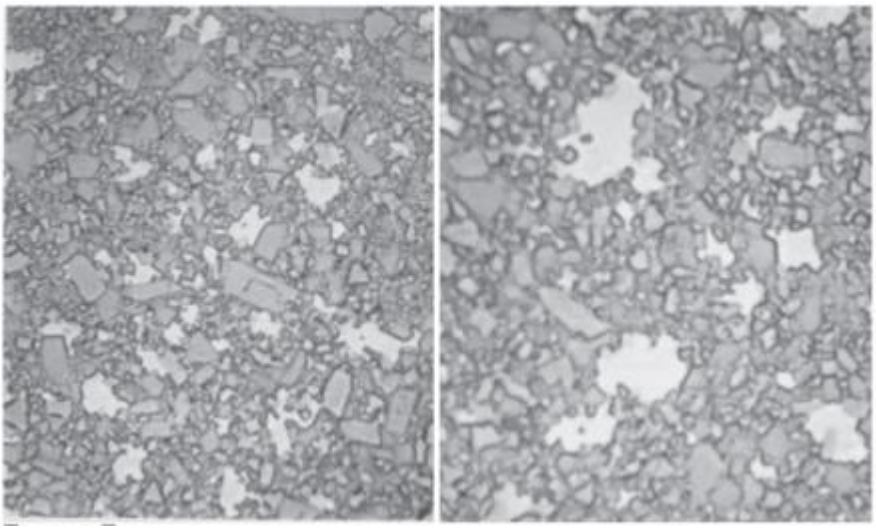


FIG. 6 SMALL ETA-PHASE PARTICLES (LIGHT GREY, ABOUT 5 µm IN DIAMETER)
(THIS FIG.6 IS REPRODUCED FROM ISO 4499-4)

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Powder Metallurgical Materials and Products Sectional Committee, MTD 25

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
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